

# THE JOURNAL

OF THE



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# THE JOURNAL

*of the*

## AMERICAN ASSOCIATION *of* CEREAL CHEMISTS

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Vol. I.

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No. 2

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**A**TENTION Brothers: The last meeting of the A. A. C. C. hasn't been held yet, not by a long shot. There will be one in St. Louis on the following dates, May 29th and 30th 1916. Will you be there? Good! We will meet you, then, at the Planters' Hotel about the 29th of May.

Make your plans now and provide that they will not miscarry. Speak to the proper authority now and get permission to be with the rest of us on the 29th. Don't be afraid of your employer, he is a live wire or he wouldn't have you around the place. Besides, he will get his money's worth no matter how he views it. You cannot get out and rub up against the other fellows without getting some new ideas and developing a determination to apply some new thoughts to the situation at the plant. And right there is where the investment pays. Pays your employer in cash and pays you in mental development.

Come loaded with a bunch of convictions and prepared to back them

to the last ditch. Remember, the majority rules in this association and your vote is as good as the other fellow's. At the meeting held in Wichita in February there was a lively time and the members that were not present missed a fine chance to have their brain cells agitated. It was pretty evident that we all had settled convictions and were so well satisfied with them that we had not taken the time to look elsewhere for something better.

It is truly remarkable how seldom two or more chemists do a thing in the same manner. Every one has been brought up to do each of the many determinations in the way that his instructor *thought* best, only to find out after getting away from the influence of the teacher that there were many modifications of each method. Then when thrown on his own resources he promptly goes to work and establishes a short cut and a variation or two. The result, every method in use in the cereal laboratory is so disguised by modifications that it is

difficult to find two operators using the same combination.

There is a danger in having more than one permissible method for any determination. If there are two methods of equal merit, then let the most economical one be the official one. And in this connection let it be remembered that in the busy laboratory the economy of time is fully as important as economy in the expenditure of money. With many such factors to be considered, it is not at all surprising that at the meeting in February it was most difficult to arrive at any common point from which we could start to work out a method that would be adaptable in each laboratory. *The proposed methods adopted were arrived at not because the members were united in favoring them, but to form a basis for all to work from, in an effort to arrive at the best.* It might be mentioned here that there has been considerable criticism of all that was done at that time, but that, far from being discouraging, is the best part of all. It has made us all think and investigate. Elsewhere in this issue there will be found comments, caustic and otherwise, concerning the proposed methods. Good! That is more than we could get by writing letters.

Following the winter meeting, our President sent out samples of flour to each of the members and to such non-members as desired, for the purpose of getting an idea as to the working value of the new methods. All the reports are in but can not be included in this number for want of space. They will come in for a thorough discussion at the next meeting.

In about a month we will meet again, and this time for the first time following a period of effort by each member along lines similar to those followed by all the other members. The results from the combined efforts of the entire association concentrated on a few lines of effort should produce results that will lead to a satisfactory solution of some of the problems.

However, it will depend on you, Mr. Member, to get in and do your share before the meeting. This is the time of the year that you will have the spare time for research if you will ever have it; in a couple of months there will be a new wheat crop and a great amount of wheat inspection that will require every minute of the long day. There will be soundness tests, strength tests, milling tests, and many other tests to occupy the time and there will be no time left for pure investigation. Now right here is where the value of the standardization of testing will work out to the good of all. If we all use the same methods in making these tests, there will be good reason to suspect that the conclusions will all be alike, and that is what they should be. When such is the case our employers and clients will feel that they are getting their money's worth when they ask for the dope on whatever variety of material they submit for analysis. Have you thought of that? Do you think that the value that you get for the gliadin number on a sample of flour is the same that some other laboratory will get on the same sample? The chances are pretty good that if the other fellow followed the same procedure that you did, that his results would be reasonably close to yours. That will be an improvement over the present conditions, most certainly. Be prepared to help.

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## PROGRAM

Discussion under the following heads will occur beginning with the meeting on the morning of Monday the 29th of May.

**METHOD FOR TOTAL SOLIDS:** The factors governing the use of the proposed method as it stands are to be considered and discussed as to their bearing on the operation. The elements of time, temperature, concentration, filtration, etc., will all be gone over carefully.

**METHOD FOR REDUCING SUGARS:** There has been objection to the proposed method for the determination of sugar, based on the grounds of its limitations for accurate work. The proposed temperature is thought to be too high by some and the determination in the unclarified solution by a gravimetric method basing the calculations on the cuprous oxide, has provided others with cause for dissatisfaction. The proposal to base calculations on the cupric oxide will be considered, as well as the desirability of clarifying.

**METHOD FOR DET. OF ACIDITY:** The methods as proposed seems to give satisfaction but time will be given to the consideration of any complaints that may be entered.

**METHOD FOR DET. OF SOLUBLE PROTEIN:** The determination of the soluble protein in the solution prepared for the determination of the other factors may be open to criticism and time will be provided for consideration of this feature.

**BAKING FORMULA:** A full and free discussion of the proposed baking formula will be provided and the members are invited to suggest improvements. The proposed formula has been freely criticised and some claims against it are most certainly well founded.

**TYPE OF LOAF:** This matter, neglected before, will receive careful consideration this time and members are urged to try out the different styles of pans and the different ways of shaping the loaf. Will we bake a single or a double loaf, etc.?

All these things must be settled some time and the members are urged to do investigating along each line and preserve their data to be used in making conclusions relative to the disputed points. Only an actual comparison of the methods in use will serve to bring out the weak points in each. We want speed, speed enough at least, to give commercial value to the tests, but first must come *accuracy*,

also last and all the time in between, *accuracy*.

It is expected that several of the members will have papers to read at this meeting and it is suggested that they notify the President on or before the 25th of May, so that the necessary time will be provided. The discussions and the papers will be heard at the Monday meetings and the Tuesday morning meeting. Tuesday afternoon will be preserved for the election of officers, the consideration of a proposed amendment to the constitution, and the adoption of some plan that will provide a satisfactory means of financing THE JOURNAL.

If agreement can be gotten in the consideration of one or more of the methods mentioned above, it will mean much toward standardizing the laboratory work and the faster that the best methods are recognized the quicker will the work of the chemist be made to serve the manufacturer in the best way possible.

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#### MINUTES OF MEETING OF THE AMERICAN ASSOCIATION OF CEREAL CHEMISTS, WICHITA, KANSAS, KANSAS CLUB ROOMS FEBRUARY 4 and 5, 1916.

Meeting opened by Pres. Weaver  
at 2:00 P. M., February 4th.

The first business was the election of the following new members:

C. R. Southwell, Wichita Flour Mills Co.,  
Wichita, Kans.

Ralph Potts, Kansas Milling Co., Wichita,  
Kans.

D. S. Jackman, Kansas Milling Co.,  
Wichita, Kans.

Leslie R. Olsen, Royal Milling Co., Great  
Falls, Mont.

F. H. Loomis, Wichita Milling & Elevator  
Co., Wichita Falls, Tex.

Gordon Auld, Wells-Abbott-Nieman Co.,  
Schuyler, Nebr.

The members present:

R. Wallace Mitchell, A. W. Estabrook,  
C. J. Patterson, J. M. Hogan, A. R. Sasse,  
H. E. Weaver, E. G. Wahlin, C. F. Buck,  
P. M. Patterson, F. B. Porter, Ralph Potts,  
C. R. Southwell, D. S. Jackman.



Mr. Weaver followed the election by a short speech of welcome to the new members and gave an outline of the work before the Association. The meeting was then opened for business relative to methods.

R. W. Mitchell reported on investigation covering moisture determination, with the collaboration of E. G. Wahlin. Upon their recommendation, the following moisture dish was accepted as the standard form, the dishes to be made for the Association:

The moisture dish shall be one of rigid construction, of glass or metal, preferably aluminum, and provided with a close-fitting cover. It shall be of low form, corresponding to approximately the following specifications:

Straight sides, diameter  $1\frac{1}{2}$  inches or greater.

Depth not greater than 1 inch.

Next followed a report on Crude Fibre determinations by C. J. Patterson with discussions on the one and two filtration methods.

Mr. A. W. Estabrook read a paper on the subject of "The Nitrogen Determination as Applied to Cereals." Following Mr. Estabrook's paper the meeting was adjourned to the banquet room where dinner was served. Following the dinner we attended the theatre in a body.

Saturday morning session called at 9:00 o'clock.

Letters from J. E. Wihlfahrt and R. M. French were read, both expressing their regret that they were not able because of circumstances to attend the meeting.

Treasurer's report read and motion made and carried that it be accepted as read.

A general discussion followed relative to the determination of "gluten" in wheat. On the suggestion of Mr. C. J. Patterson the following resolution was presented and adopted:

WHEREAS, In view of the fact that the nitrogenous body found in wheat and flour, and commonly called gluten, is without any definite meaning, owing to impurities present in the washed out mass, and whereas, there is no accurate means of arriving at the quantity of so-called gluten;

RESOLVED, That this Association wishes to discontinue the use of the term "gluten," and to express the nitrogenous value of wheat and flour as protein, calculated from the total nitrogen, times the factor 5.7. When made on wheat, to be on whole wheat meal.

Mr. A. R. Sasse read a report on investigation on acidity, water soluble solids and reducing sugars.

P. M. Patterson then spoke on the distillation of N H 3 from flour and wheat meal without digestion, but in presence of Na O H. C. J. Patterson and P. M. Patterson appointed to investigate.

A general discussion on Baking Methods followed. C. J. Patterson suggested the adoption of a uniform formula, pan and method of reporting baking tests; the following formula being elected as proposed:

Flour .....	392	grams
Sugar .....	7	grams
Salt .....	5	grams
Yeast .....	5	grams
Lard .....	5.5	grams

Those members using mechanical mixers, to use a proportionate amount of flour and other ingredients, so as to have dough enough to scale on a basis of 392 grams of flour before fermenting. Official pan to be adopted later, after investigation by all members.

Motion made to report volume in cubic inches. Motion carried.

The following appointments were made by Pres. Weaver, to put before the various Boards of Trade the Association's Resolution on Protein, to be adopted by grain brokers and millers alike:

Wichita, Mitchell, Southwell.  
Oklahoma City, E. G. Wahlin.  
Kansas City, Estabrook, Hogan, Sasse, Patterson.  
Hutchinson, Weaver, Patterson.  
Fort Worth, F. B. Porter.  
Omaha, Estabrook.  
St. Louis, Estabrook.

A copy of the resolution relative to protein to be sent to all the trade journals.

Moved, seconded, and carried that we extend a vote of thanks to the

Kansas Club for the courtesy of the use of their rooms.

Meeting adjourned by President Weaver.

## METHODS

At the present time the approved and the proposed methods of the Association are as follows:

### APPROVED:

**MOISTURE:** Dry a suitable sample in an oven at the temperature of 101 to 105 degrees C. until the sample ceases to loose weight.

**ASH:** The official method of the A. O. A. C.

**PROTEIN IN FLOUR:** Nitrogen to be determined by either the "Kjeldahl," "Gunning," or "Kjeldahl-Gunning-Arnold" method. The calculation of the "Protein" to be with the factor 5.7.

**ACIDITY:** The official method of the A. O. A. C.

**WATER-SOLUBLE SOLIDS:** The official method of the A. O. A. C.

**REDUCING SUGARS:** The official method of the A. O. A. C.

**LOAF VOLUME:** The loaf volume to be reported in terms of cubic inches.

### PROPOSED:

**PROTEIN OF WHEAT:** The protein of wheat to be determined on the fine meal of the whole berry by one of the methods as provided in the determination of protein. The calculation to be made from the total nitrogen using the factor 5.7, and reported as "Protein."

**WATER-SOLUBLE SOLIDS, REDUCING SUGARS, AND ACIDITY:** 25. grams of the sample of flour are shaken thoroughly with 250 cc. of distilled water, free of carbon dioxide. Digest for one hour at 40. degrees C., shaking every fifteen minutes; centrifuge and filter. Titrate 50. cc. of this filtrate with N-20 alkali, using 2. cc. of carefully neutralized Phenolphthalein as indicator. Each cc. of N-20 alkali will equal .09% acidity as Lactic acid.

Total water-soluble solids determined on 25. cc. aliquot of the centrifuged solution by evaporation to dryness in a tared dish, and dried to constant weight at 100. degrees C.

**REDUCING SUGARS AS CRYSTALLIZED MALTOSE:** Use 10. cc. of the prepared solution without further clarification. Determine sugar according to the method of Munsen and Walker. (Bul. 107 appendix). Calculate the result to crystalized maltose.

**CRUDE FIBER:** The fat-free material (bran or other feed products) is carefully brushed into a lipless liter beaker or Erlenmeyer flask and 200 cc. of boiling 1.25% sulphuric acid is added, then boil for 30 minutes using a suitable condenser. At the end of this time, 200 cc. of a 3.52% solution of Na OH is added and the whole boiled another thirty minutes. The solution is then filtered as quickly as possible through alundum crucible of the grade known as "Maximum Porosity," washed neutral with distilled water, then washed with alcohol and ether, and dried till weight is constant. Incinerate, cool and weigh; the loss in weight on incinerating is the weight of crude fiber.

**COLOR SCALE FOR BREAD COMPARISON:** Color to be reported in the following terms, the words preferred to the numbers:

Patent	100. equalling.....	Excellent
	99. equalling.....	Normal or Good
	98. equalling.....	Poor
Straight	96. equalling.....	Excellent
	95. equalling.....	Normal or Good
	94. equalling.....	Poor
Clear	90. equalling.....	Excellent
	89. equalling.....	Normal or Good
	88. equalling.....	Poor

### BREAD FORMULA:

Flour.....	392. gms.
Sugar.....	7. gms.
Salt.....	5. gms.
Yeast.....	5. gms.
Lard.....	5.5 gms.

Fermentation period to be between four and five and a half hours, with thirty minutes proof. Where a mechanical mixer is used, a proportionate

amount of all the ingredients is used so that dough will be had to scale on the basis of 392 gms. of flour. Dough to be scaled before fermentation.

### NITROGEN DETERMINATION ON CEREALS

Paper read at February meeting:

During the last few years the writer has been called upon to make a large number of protein tests on wheat and wheat products and with the increasing number it has been necessary to study ways of making these tests more rapidly and at the same time with a higher degree of accuracy.

Perhaps these methods and the apparatus may not be new to all of you but by going into detail, it is very possible each of you may take away with you some new ideas. In describing these tests, let us use wheat as the substance we are testing and what applies to wheat in general will apply to the products made from it. I have always regarded the Gunning modification of the Kjeldahl method as the simplest and best method of getting the nitrogen of food products and this is the method which is followed in our laboratory in making protein tests on wheat. The sample of wheat is tested just in the condition that it arrives at the laboratory, that is, without removing any foreign material or without calculating to a moisture free basis.

The sample is ground to such a degree of fineness that it will pass through a No. 30 grit gauze. The grinding is done on a small coffee or spice mill to which we have attached a pulley and which we drive from a motor. A machine like this is always susceptible to an error caused by a certain amount of material remaining in the burrs or on shelving places in the machine.

In grinding a very small sample, enough material might become mixed

from a former sample to cause variation in the results. In grinding a very small sample the machine can be opened and cleaned easily before grinding, but in ordinary practice where we have plenty of wheat I usually consider it sufficient to run a handfull of wheat through the machine and discard that portion before grinding a portion for the test. In grinding a large number of samples which usually come to us in envelopes or in wooden trays we usually immediately transfer the ground portion to the container in which the original wheat came. This saves time and avoids possibility of error in the marks of the sample of wheat.

In weighing a sample for a protein test we are careful to stir the sample thoroughly getting the finer portion mixed with the coarser and we weigh for this test exactly 1 gram of material. In weighing this material it is not absolutely necessary to use the extreme accuracy of the analytical balance, as compared with this accuracy the accuracy of the Titration with n-10 solution is very crude and I have found it a waste of time to wait too long for the balance to swing or come to rest.

For the digestion we use the 500 cc. Kjeldahl flask into which we weigh the 1 gram of wheat and with a small dipper, approximately 10 grs. of Potassium Sulphate and with a pipette about 12 cc. of sulphuric acid. We have adjusted these quantities of potassium sulphate and acid to such a point that they will digest at the most rapid rate and at the same time allow the digested substance to remain in a semi-liquid state at the end of the process. If the amount of acid be increased or the potassium sulphate diminished the digestion will proceed much more slowly while if the acid be diminished or the potassium sulphate increased, the digestion will proceed much more rapidly, but the digested substance will become very hard in the flask and will occupy some time in dissolving.



This digestion we conduct with the flask on its side with the neck inserted in a hole in a lead pipe, the flask having just enough pitch to allow the condensing fumes to run back into the flask freely. The flask is heated directly under the center and by a flame as evenly over the surface as possible and extending about half way up the side of the flask. We have found that by exposing the flask to the naked flame with as little obstruction as possible between the flame and the flask that there is less breakage of flasks and a considerable hastening of the digesting process. The lead tube into which the flask extends is connected to the outer atmosphere by a lead pipe flue which usually effectually carries off the fumes by natural draft. There are times at which it will not do this and we have this flue connected to an exhaust fan driven by a motor which draws off the fumes by means of an air jet. This scheme of exhausting the fumes has proved very satisfactory.

The distillation process is conducted by first dissolving the digested material in the flask by filling the flask about half full of hydrant water, adding a small piece of pumice stone to prevent bumping, a few drops of phenolphthalein indicator and enough sodium hydroxide to make the solution alkaline. The delivery tube of the still is immersed in the equivalent of 25 cc.'s of N-10 sulphuric acid. The flask is attached to the still and a burner lighted immediately. The attaching of the flask to the still should be done very quickly after neutralizing it and should not be done before the digested substance is entirely dissolved nor before it is thoroughly neutralized. If the flame is not lighted immediately, the cooling of the contents of the flask may cause it to draw from the N-10 acid from the receiving bottle.

During a recent cold spell we found on one occasion that we could not run the usual amount of water through our still because the intense cold of the

water caused condensation so rapidly that it drew over the contents of all our receiving bottles. It is well to watch your flask as it begins to boil as this is the point at which there is the greatest danger of foaming and an alkaline material getting over into the distillate. We have had considerable trouble at different times with flasks foaming so much in boiling that it spoiled our results and we have investigated this subject very thoroughly so that now we are bothered with it very little. A flask that is filled too full will cause the boiling to force the liquid up the neck of the flask. We formerly used zinc to prevent bumping, and if placed in the flask before neutralizing, the hydrogen gas given off tends to make the liquid foam when it begins to boil, but perhaps the most troublesome cause of foaming in the flasks is due to carbonate in the sodium hydroxide.

We are now dissolving our sodium hydroxide in a large jar and drawing it off by a faucet which is placed two inches above the bottom. This allows the almost insoluble carbonate and any foreign material to settle to the bottom and we draw off only the clear hydroxide solution. Since taking these precautions we have been almost entirely free from troublesome foaming. We usually distill over about 150 cc. and before turning out the flame we drop the receiving bottle so that the delivery tube is above the surface of the liquid and allow it to run this way for a few minutes. This very effectually washes down into the bottle any of the distillate which may have been clinging to the inside of the tube.

We have built for our use a still made of iron pipe in the form of a series of Liebig condensers connected up with each other so that we can turn the valve and allow a one inch stream of water to flow through the whole series of twenty-four sections. The delivery tubes of the stills are of block tin and are bent to go through the stopper into the distilling flask and

without a joint of any kind reach into the receiving bottle. The flask is supported over the flame by a steel wire ring which is adjusted by a set screw attached to the Bunsen burner.

The titration is made with the flask on a white surface facing a good light. The indicator usually recommended is cochineal. My experience with this indicator is that usually the end point is not distinct and sometimes so indefinite as to be of no value at all. At a few other times, however, the end point is very distinct and in this case the alkaline color of the indicator is a true violet color as described in the text book on this subject. Using cochineal on a blank determination, that is on water only, the end is very distinct and the alkaline color a true violet, but on a Kjeldahl distillate it rarely gives either result. A member of this Association gave me a hint that has helped me get from cochineal the violet color and a sharp end point. If a very small portion of an iron salt be added to the distillate (say about 1-100 of 1%) the end point will be sharp and the alkaline color a deep violet. I have tried this out perhaps a dozen times and it has not failed yet.

Sodium-Alizarine-Sulfonate has given us a very sharp end point and seems to be perfectly reliable as an indicator for ammonia. Methyl orange is also very reliable. These three indicators are all permissible to use in this titration, but I think that the best results can be gotten with the sodium-alizarine-sulfonate.

In this work I use N-10 solutions. These I standardize by titrating the alkaline solution against potassium bitartrate purified according to the U. S. Pharmacopeia. The Na O H solution I make absolutely standard and the acid solution only approximately standard and of known normality.

We weight exactly one gram of material, use 25 cc. of N-10 acid and have a table that shows protein directly from the burette reading.

This saves time in figuring a large number of tests. The whole nitrogen process we can complete if necessary in about forty minutes.

I will mention a few things related to this work. In getting sulphuric acid from a carboy we draw it off by exhausting the small bottle with our bunsen pump and thus drawing the acid through a glass tube from the carboy to the smaller bottle. It takes about three minutes to fill a nine pound bottle.

I have noticed that sometimes when the digested substance becomes very dry that no ammonia comes over during the distillation. I have not yet been able to find just what becomes of the ammonia in this event and should like some information on this point.

I have noticed that when a flask first begins to boil that a smoky looking gas comes over into the distillate and results are not good when this occurs. If others have had this experience, I should be glad to hear from you.

The condition that exists between the acid and the ammonia in the distillate has long been of interest to me. A definite chemical evidently is not formed in solution because upon standing the ammonia will leave and upon boiling it can be driven off in a few minutes. I have tried using only water in the receiving bottle thinking that it would do as well as acid but have always found that this resulted in the loss of some ammonia.

A. W. ESTABROOK,  
710 Wyandotte St., Kansas City, Mo.

#### THE OFFICIAL METHOD FOR THE DETERMINATION OF CRUDE FIBRE AS APPLIED TO PURE WHEAT BRAN.

Paper read at February meeting

The official method of analysis (U. S. Bureau of Chemistry, Bul. 107) for determining crude fibre has been criticised by many chemists.

The source of error seems to lie with

the different filtering materials used. These being linen, asbestos, glasswool, sand, alundum crucibles, and the best grade of filter paper.

The linen is used by one or two laboratories of my knowledge, with suction in Buchner funnels. It would be hard to duplicate the weave of linen, and this naturally would cause some variations. However this method has given satisfaction as far as uniform results are concerned.

A neat method for upward suction has been suggested by Pickel\*.

Forbes and Menching† have suggested the use of fine sand. This method is not of value, as the results are not satisfactory and the time for filtration even with suction is too long.

Sweeney‡ proposes to omit the first filtration, and neutralize the acid with a known amount of sodium hydroxide, adding the alkali to the same vessel and filtering through asbestos. Kennedy\*† has modified this slightly, and this method has been followed out in this laboratory. The results are slightly higher with the asbestos filter than with the alundum crucible.

The main hope of my investigations on crude fibre is to find a filtering medium that will be consistent in obtaining uniform results, at the same time to increase the speed of the determination and not to change the principles of the official method.

The Kennedy modification of the Sweeney method has been used in comparison with the official method.

Crude Fibre Method: The dry

fat-free residue is carefully brushed from a small paper extraction thimble into a lipless 1000 cc. Erlenmeyer flask. Two hundred cc. of boiling 1.25% sulphuric acid are added and the mixture boiled for thirty minutes, using a reflux condenser at the end of this time two hundred cc. of boiling 3.52% Na O H are added and boiled for thirty minutes.

Sweeney has proposed a Na O H solution of another strength to neutralize the acid, using an indicator.

A number of titrations have been made with the 3.52% solution and shows the alkaline solution to vary from 1.20% to 1.27% Na O H. This of course does away with another solution, the care in neutralizing, and most of all saves time.

A Buchner funnel is prepared with a S. & S. 595 filter paper. Care must be taken that there are no open channels around the edge.

After the final boiling the fibre is filtered off rapidly, and washed neutral. From here it is transferred to an alundum crucible and washed with alcohol and ether. Placed in a drying oven at 100 degrees C., then cooled and weighed. Repeat the drying and when the weight is constant incinerated for 15 minutes to 30 minutes at low red heat or until a gray ash is obtained.

All results are calculated to the original amount of material used.

C. K. Francis\* has shown in recent work on asbestos for crude fibre determination that asbestos loses weight on being ignited.

\*Journal of Ind. Eng. Chem. Vol. 7, No. 8 (1915)

Table No. 1. Crude fibre determined by the one filtration method and the official method using different filtering mediums:

	Linen(420)	Paper	Alundum	Asbestos
One Filtration Method.....	8.70	9.00	9.13	9.60
	8.90	9.40	9.34	9.72
	8.50	9.32	9.20	9.52
Official Method.....	8.95	9.17	9.40	9.80
	8.70	9.27	9.26	9.76
	8.65	9.09	9.22	9.55

\*Journal of Am. Chem. Soc. No. 2 (1910) 280.

†Journal of Am. Chem. Soc. No. 5 (1913) 258.

‡U. S. Bureau of Chem. Bul. 137 (1910) 157.

\*†Journal of Am. Chem. Soc. 4 (1912) 600.

The fibrous material of bran and other wheat products is of such a nature that it should be incinerated in a short length of time. Further heating causes varying results, which, no doubt is due to volatile salts contained therein.

C. K. Francis also has shown the amount of coarse and fine filter paper digested by acid alone, alkali alone and both acid and alkali together. Either one alone digests only a very small quantity, while both together digest 11.64% of coarse and 10.77% of fine.

Table No. 1 shows the results obtained by using the different filtering mediums, which do not vary except with the asbestos and linen. This is no doubt due to the loss in weight by the ignition of the asbestos in the first case, and small particles passing through the linen in the second.

This table also shows results obtained by the one filtration method and the official method.

#### Summary:

1. A method is proposed for the determination of crude fibre in bran, and other wheat products, in which alundum crucibles are to be used as the filtering medium.

2. No difference in results have been obtained between the one filtration method and the official method.

3. Slightly lower results were obtained with linen.

4. Asbestos gives high results due to loss in weight on ignition.

C. J. PATTERSON,  
Ismert-Hincke Laboratory.

### A FLOUR STORAGE EXPERIMENT

A quantity of flour made from an average mixture of Kansas wheat was divided into two portions and preserved under different storage conditions for a period of two weeks. The test was made to determine what effect the warmer and more humid weather

of the spring months would have on the keeping qualities of the flour.

Sample number one was stored in a dry place at a temperature of from 65 degrees to 70 degrees. Sample number two was kept in an atmosphere slightly more humid and at a temperature of 80 to 85 degrees.

After two weeks the flours were again analysed for soluble solids, soluble protein, and acidity. Gluten balls were made and allowed to soak in water at a temperature of 65 degrees F. until they had fermented sufficiently to lose their firm consistency, and rise to the top of the water. The time was noted when the ball softened and also when it came to the top.

Baking tests were made on the original sample and on samples No. 1 and No. 2 after storage. The baked loaves of the fresh flour and of No. 1 were the same, no difference was noticeable. The loaf from No. 2 was 14 cubic inches larger than the other loaves and considerably whiter. The texture of the loaf from No. 2 was very inferior to the others, it being coarse and blubbery, there was also noticeable a disagreeable odor on the warm loaf.

The data on the water soluble factors was worthy of notice and is here given:

Original Sample	No. 1	No. 2
Water sol. solids . . . . .	9.34%	11.68%
Water sol. protein . . . . .	2.18%	3.24%
Acidity . . . . .	.094%	.132%

The soaked gluten balls were allowed to stand in water at 65 degrees F. and their behavior noted.

Gluten ball from fresh flour was still firm after 28 hours, showing good condition.

Gluten ball from sample No. 1 was still firm after twenty-four hours, but had begun to soften at twenty-eight hours, and was floating at thirty-two hours.

Gluten ball from sample No. 2 was soft in eight and a half hours, and was found floating at twelve hours.

The conclusion drawn from these tests was that it would be unwise to



store flour during the warm weather, except under cool and dry conditions.

P. M. PATTERSON,  
Millers' Laboratories,  
Hutchinson, Kansas.

### REPORT ON MOISTURE DETERMINATIONS

I have done considerable work along the line suggested by our worthy President at the last meeting of the Association. Namely, the comparison of the covered and the uncovered vessels in common use in cereal laboratories for the determination of moisture. The small open dish holding from five to ten grams similar to a milk dish, and larger ones holding as much as twenty-five grams of a heavy material (such as flour), were compared with glass and metal dishes provided with covers, the latter holding ten to fifteen grams of flour. For the sake of completeness, some work was done with the double wall flask used in a Brown-Duvel Grain Moisture Tester.

The following types of vessels were compared:

- (1) Aluminum pans  $\frac{1}{2}$  inch high by 3 inches diameter, no cover.
- (2) Aluminum pans 1 inch high by  $1\frac{1}{2}$  inches diameter, no cover.
- (3) Same as No. 2, but provided with cover.
- (4) Glass weighing dish  $\frac{3}{4}$  inch by  $1\frac{1}{2}$  inch with cover (straight sides.)
- (5) Glass weighing dish 2 inches by  $1\frac{1}{2}$  inches, with cover (small mough.)
- (6) Double wall Drown-Duvel copper flour-testing flask.

The large pans, No. 1, were tared on ordinary small trip scales and 25 gms. of flour used. The scales were sensitive to a twentieth of a gram and the beam graduated to tenth grams. In some cases the sample was weighed before cooling and in other cases it was cooled in the dessicator before the final weighing. This method is not to be recommended as will be seen by the results in the table below.

Vessels as described under numbers (2), (3), (4), (5), were weighed on the

analytical balances and no determinations are recorded in which the approximate weight was not known beforehand. Thus the time on the balances was about the same in all cases and the conditions were the same as hold good in practice where the operator can estimate to within several milligrams of the weight and place the weights on the pan before removing the dish from the dessicator.

The temperature of the drying oven was held at all times between 100. C. and 105. C., the desire being to hold the temperature as near 103. C. as possible. The time for drying depended somewhat on the type of dish and varied from three hours to six. I would not be able to recommend less than three hours for any practical type of dish using 10. gms. of flour. Less than ten grams increases the opportunity for error and has no commensurate advantage that I know of. It was found that samples in the tall dishes of the type of No. 4 did not reach perfect dryness in as short a time as did those in the more flat style. Thus the element of time would induce a preference for a low, large mouthed dish.

As regards the advantage of the covered dish over the dish without a cover, it begins when the sample is being weighed out; fresh flour having from twelve to fifteen per cent moisture will lose its moisture very fast when exposed to the atmosphere. Thus if the bulk of the sample is placed in the dish and the cover placed on it in an inverted position, the final balance may be made without exposing all of the sample to the air of the room and the opportunity to evaporate.

The next place that the difference is noticed is when the final weighing takes place. In case there are duplicates in the dessicator and the dessicator is opened several times between the weighing of the duplicates, there will almost always be a disagreement, with the last one weighed showing the least



moisture, when the dishes are not covered. In view of the fact that with duplicates the exact weights can be nearly anticipated the loss would seem to be in the dessicator due to the frequent opening that is necessary in handling a large number of determinations. Of course added to this error is that due to the change that begins to take place as soon as the dish is exposed due to the extremely hygroscopic nature of moisture free flour. My experience as will be noted from the table below, is, that with the covered dishes a number of dishes may be cooled in a dessicator at the same time and that the number of times the dessicator is opened or a slight delay in completing the weighing will not affect the results, but that with open dishes there is liability of error in those weighed last.

I have not been able to get results with the double walled flour testing flask that are either reliable or concordant. It has been handled exactly as provided in the circular issued by the government, using a high grade engine oil of Sp. Gr. of about .920. The thermometer used is one of medium grade having an error at 190. C. of not more than .1 C. The results as can be seen are as a rule from .1% to 1.5% from those arrived at by the method of drying. I feel that the opportunity for error due to causes other than carelessness or incompetency on the part of the operator are greater than with the method of drying and for that reason would not be able to recommend it to the society for adoption as standard.

This then is my decision as one member of the committee appointed

MOISTURE DETERMINATIONS, TABLE No. 1

Sample *No.	Dish No. 1 Open Per cent	Dish No. 2 Open Per cent	Dish No. 3 Covered Per cent	Dish No. 4 Covered Per cent	Dish No. 5 Covered Per cent	Dish No. 6 Per cent
1.....	13.9	13.75	13.82	.....	.....	.....
2.....	13.5	13.2	13.2	.....	.....	.....
3.....	.....	.....	.....	13.77	.....	13.1
4.....	13.28	13.0	13.28	.....	.....	13.1
5.....	.....	12.63	12.89	.....	.....	.....
.....	.....	.....	12.69	.....	.....	.....
.....	.....	.....	12.79	.....	.....	12.3
6.....	.....	.....	12.94	.....	.....	.....
.....	.....	.....	12.99	.....	.....	.....
7.....	.....	.....	13.26	.....	.....	.....
.....	.....	.....	13.34	.....	.....	.....
.....	.....	.....	13.33	.....	.....	.....
8.....	13.4	.....	13.14	.....	.....	12.8
.....	.....	.....	13.21	.....	.....	.....
.....	.....	.....	13.26	.....	.....	.....
9.....	.....	.....	13.08	.....	.....	.....
.....	.....	.....	13.07	.....	.....	.....
10.....	.....	13.55	13.76	.....	.....	13.4
.....	.....	13.62	.....	.....	.....	.....
11.....	.....	13.68	13.77	13.9	.....	13.2
.....	.....	13.43	.....	.....	.....	.....
12.....	.....	13.06	13.41	.....	.....	13.2
13.....	.....	12.05	.....	12.12	.....	11.4
.....	.....	12.13	.....	.....	.....	.....
.....	.....	12.08	.....	.....	.....	.....
14.....	.....	12.84	12.77	12.59	.....	.....
.....	.....	12.67	.....	.....	.....	.....
15.....	12.36	12.1	12.08	12.19	11.36	(open)
.....	.....	12.09	.....	.....	11.53	(closed)

to investigate the subject: The moisture in cereal products should be determined on a sample of not less than five grams by drying at a temperature not to exceed 105° C. until there is no more loss in weight; the vessel for the operation to be of metal or glass and provided with a cover which shall be in place during cooling in the dessicator and during the final weighing.

R. WALLACE MITCHELL,  
E. G. WAHLIN, concurring.

### SOME TESTS ON DAMAGED WHEAT

During the present year a great opportunity has presented itself for the study of various kinds of damaged

wheat. These wheats divide themselves naturally into three classes: Sprouted wheat, bin burned wheat, and what is commonly termed mow burned wheat.

Sprouted wheat is easily recognized by its sprouted and swollen condition.

Bin burned wheat is a dark mahogany color. The worst specimens show dark and fibrous when cut in two, some are very close in appearance to wheat which has been tested for moisture in the Brown-Duvel Moisture Tester.

Mow burned wheat is light grey in color, usually a little sour to the taste, and has a partly glassy and partly soft texture inside.

An analysis of these various wheats is given in the following table compared to a sample of sound wheat:

Kind of Wheat	Per cent Protein	Per cent Ash	Per cent Mois.	Per cent		Per cent		Per cent Crude Fiber
				Solu. Solids	Per cent Maltose	Sol. Prot.	Per cent Acid	
No. 1 Hard (Dark)	12.52	1.75	12.40	4.88	1.82	2.12	.180	2.48
Sprouted Wheat . . .	10.04	1.86	14.80	8.84	5.13	4.08	.630	....
Bin-Burned Wheat .	12.52	2.12	13.20	4.34	1.63	2.48	.540	3.92
Mow-burned Wheat	11.16	1.82	12.60	6.52	3.98	3.80	.530	3.00

In making this analysis the different kinds of wheat were separated from all others as nearly as possible.

The per cent protein given has no significance as they merely vary as the wheats from which the samples were selected.

It will be noticed that the greatest point of difference for the sprouted wheat is increased "soluble solids," "maltose," "soluble protein," and "acidity."

The bin burned wheat shows decided increase in ash and crude fiber.

The mow burned wheat shows some increase in all the determinations.

Upon drying and storing, all the samples with the exception of the sound wheat, show a decided loss in acidity.

Baking test made from the following wheats show as follows:

Sprouted wheat (15%): A loaf of heavy, sticky body while hot, loss of color; when cool some dampness, a weak body and a very coarse texture. No loss of loaf volume.

Bin burned wheat (20%): A loaf of low volume, and dark color.

Mow burned wheat (15%): A sticky loaf same as with sprouted wheat, loss of color and poor texture. Some loss of loaf volume.

### Conclusions:

**SPROUTED WHEAT:** The chemical change during germination increases the sugars and renders the protein more soluble. The sugars give the stickiness in the loaf. The protein has become unduly weakened, and fails to give the loaf a sound body.

**BIN BURNED WHEAT:** The action during heating or burning tends to decrease the

amount of carbohydrates, thereby increasing the percentage of ash and fiber.

**MOW BURNED WHEAT:** This wheat seems to be partially burned and partially decayed. It imparts to the flour characteristics of both the sprouted and bin burned wheat.

H. E. WEAVER,  
Larabee Mfg. Co.

### OUR BAKING FORMULA

Extracts from two letters received from members of the Association are here printed to show what we are up against in preparing a formula that will give universal satisfaction. It seems to be pretty certain that we will have to change the proposed formula to some extent and it is hoped that what is found here will serve to guide us in making such changes. A careful consideration of the points brought out will serve to clear the decks of our minds for fast and accurate action when the times comes.

B. H. Kepner says:

The minutes of the last meeting of the Association came to hand a few days ago and I was greatly interested in the proceedings. I really do not think that we should attempt too much all at once. The members should appreciate the fact that the Association is new and that it will take considerable time to work out the form of methods which will be suitable for all laboratories concerned. If at the end of a year's time we have decided upon a standard method for half of the determinations which will be made on wheat and flour, we can consider that we have done exceptionally well. In a matter of this kind we must make haste slowly. I think the best plan would be to have each member send in his method for determination and then try to get a method embodying all the good features of these methods. For example, the dough formula which was adopted at the last meeting was evidently the results of one man's work with a few others favoring it.

The method of testing flour by the baking process is undoubtedly the most important test which flour chemists are called upon to make. For this reason we should be very careful about the standard formula. This formula is evidently figured out on the barrel basis. You are doubtless aware that the large bakers, at the present time, are not figuring their formulas after this method. They are using so many pounds of material to 100 pounds of flour. Figuring formulas on the percentage basis has great advantages in cutting down or increasing the size of a dough desired. The formula adopted is not true to bake-shop conditions and that I presume was the idea which was uppermost in the minds of the originators of it. We all know that the amount of sugar and lard or other enriching materials varies in the different factories. Two things that baking experts are quite agreed upon, is the amount of salt and yeast used to 100 pounds of flour. They use  $1\frac{1}{2}$  pounds of salt and 1 pound of yeast to 100 pounds of flour. For very rich bread some are using  $1\frac{1}{4}$  pounds of yeast to 100 pounds of flour but we are not interested in making rich bread. The formula as decided upon figures out about  $1\frac{1}{4}$  pounds of yeast and salt to 100 pounds of flour. For test work, we think the fermentation period extends over too long a period of time. A small amount of dough is easily affected by different atmospheric conditions. Then, too, results are required more quickly than this formula will give them. Take for example, in our own laboratory where we do the testing for seven different mills there are days upon which it is necessary to make in the neighborhood of 30 baking tests and in the Fall there are a great many more. It would be impossible to make, bake, cut and judge these loaves, made by this formula, on a short winter's day. I think the idea of getting down to a baker's formula is an excellent one, and I can see no harm in doubling the

amount of yeast and cutting down the time by a little less than one-half. The one thing we want to get away from is the dough formula which has been used to a great extent in the past. In this case a ferment is set and abnormal of yeast and sugar are used and the proof of the dough in the pans is allowed to go to the extreme with comparatively no system of turning. The times during fermentation a baker usually turns his dough depending upon the grade of flour used, are as follows: 1st turn,  $2\frac{1}{2}$  to  $3\frac{1}{2}$  hours; 2nd turn, 1 to  $1\frac{1}{2}$  hours; 3rd turn,  $\frac{1}{2}$  to 1 hour; bench,  $\frac{1}{2}$  hour; proof in pans, about 45 minutes. This system is for Manitoba Spring Wheat Flours by using  $1\frac{1}{2}\%$  salt and  $1\%$  yeast. By using  $2\%$  yeast the time of the dough is decreased by a little less than one-half. The type of the pan used for testing work can either be the low or the tall type. The dough in the tall pans requires a little more proof because it expands in only one direction. The cut loaf baked in these tall pans is somewhat easier to judge than when baked in the low ones. There is very little difference in the volume of the two. The system used in marking the samples has been placed on the percentage basis. It should have more of a spread between the different grades. The system adopted is more like the old type of a system which millers used, 1,  $1\frac{1}{2}$ , and 2. There is certainly more than a spread of 10 points between our "patent" and "clear." Mr. Jacobs has a system which is about the best one I have ever seen. By giving a high patent the value of 100 and a "clear" the value of zero, mixtures of these two flours are made varying five points from zero to 100. The comparison is then made with a sample under investigation with these standards. By using this system it is very easy to give the exact color value of a flour as compared with "patent" which has the value of 100. Some laboratories do not mark on the scale suggested. If they are

testing "clear" grade they use a "clear" standard giving the value of 100 all through. The markings for excellent, good and poor seem to me to be too close together. It would be certainly hard to convince an average mill man that his flour was poor in color if it run 98% in comparison with the standard. Like everybody else he naturally would say it is only two points off in color and therefore, cannot be very badly down. I think it would have been wise to have had more of a spread between excellent, good and poor colors. I think your decision in regard to protein is very good. The standard method for "water soluble solids," "reducing sugar," and acidity" is also very good. The temperature and the time of extraction are about the only two points which must be strictly observed in these determinations.

Then from Mr. Hogan we have:

By using the proposed formula we are going back instead of forward. About six years ago we used a formula similar to this one, not realizing the inequality of the comparison between various flours. For the sake of comparison say we have two flours, the one having 60% absorption, and the other having 62.5%. One can readily see that the flour which has the greater absorption will make more dough per hundred pounds and in like manner if we use 392 grams of flour in our formula then the sample showing 62.5% absorption will have an advantage over the other one at least in so far as volume and weight is concerned.

On the other hand if we scale off a uniform amount of dough for each loaf, regardless of the absorption, we will get more uniform results. I find that this gives a better comparison and tends to overcome the advantage that one flour may have over the other by reason of its moisture content and absorption. At the same time if the flour having the least absorption makes as large or larger loaf than the other,

it speaks for itself. It does not require the advantage over the other flour in weight of dough.

Small bake shops base all their calculations on the "bucket." The large bake shops base their calculations on the hundred pounds of flour.

The large bakeries use about  $1\frac{1}{2}$  to 2 per cent salt,  $1\frac{1}{2}$  to  $3\frac{1}{2}$  per cent of sugar, 2 per cent of lard or oil, and of yeast from  $1\frac{1}{4}$  to 2 per cent. If malt extract or condensed milk is used then the sugar is cut.

In our laboratory we would favor a formula somewhat like this:

Flour.....	600.	gms.
Sugar.....	12.	gms.
Salt.....	10.	gms.
Yeast.....	12.	gms.
Lard or oil.....	12.	gms.

Fermentation temperature of 78. F. to 80. F.

### ABSTRACTS

"Better Bread by Means of Natural Lactic Acid," by Arnold Wahl, Jr. of Ind. and Chem. Eng. 7, 9, p. 773.

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"Food Inspection Decision 160. Gluten Products and 'Diabetic' Food."

Ground gluten is the clean, sound product made from wheat flour by the almost complete removal of starch and contains not more than ten per cent (10%) of moisture, and, calculated on the water-free basis, not less than fourteen and two-tenths per cent (14.2%) of nitrogen, not more than fifteen per cent (15%) of nitrogen-free extract (using the protein factor 5.7), and not more than five and five-tenths (5.5%) per cent of starch (as determined by the diastase method).

Gluten flour is the clean, sound product made from wheat flour by the removal of a large part of the starch and contains not more than ten per cent (10%) of moisture, and, calcu-



lated on the water-free basis, not less than seven and one-tenth per cent (7.1%) of nitrogen, not more than fifty-six per cent (56%) of nitrogen-free extract (using the protein factor 5.7), and not more than forty-four per cent (44%) of starch (as determined by the diastase method).

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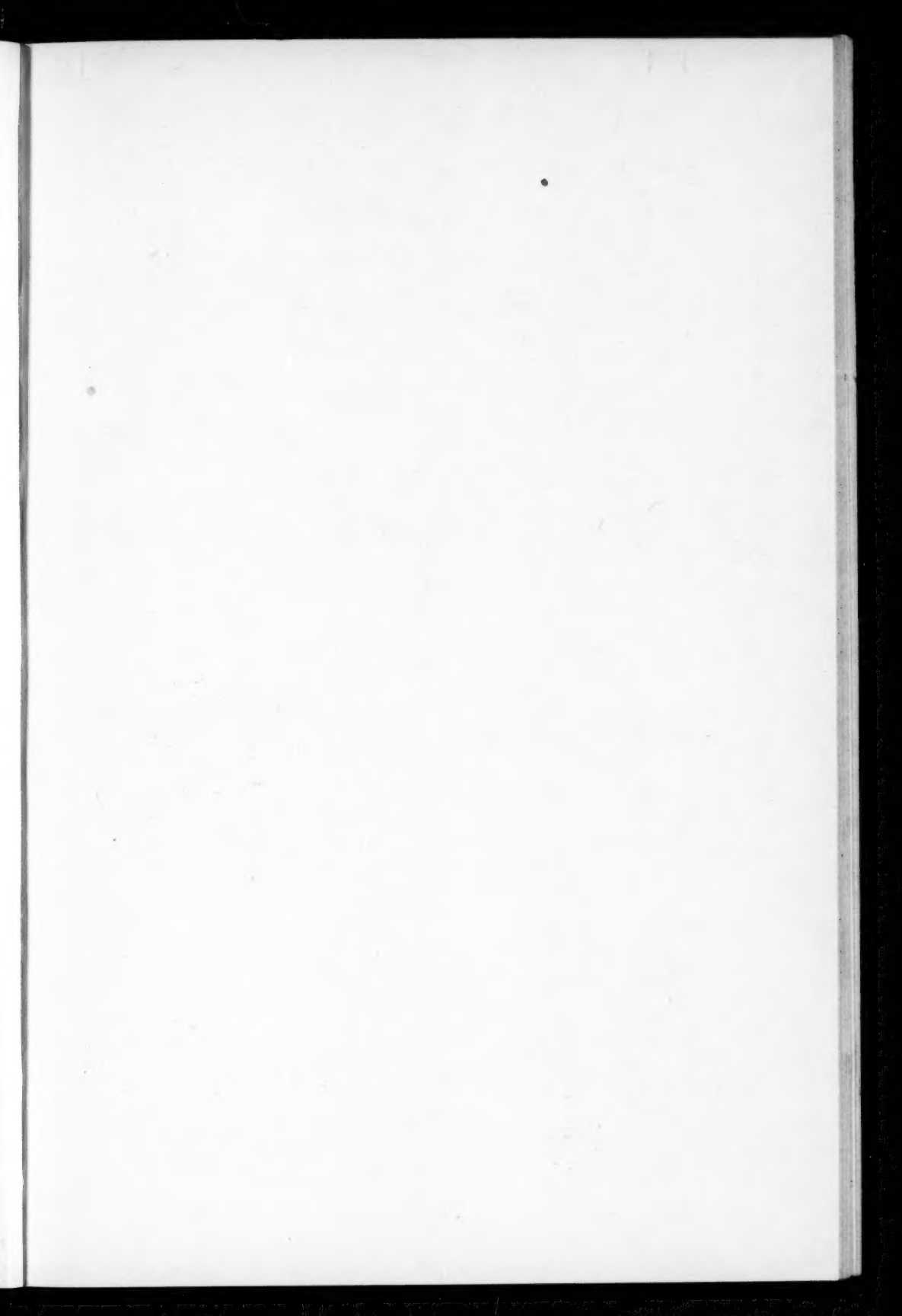
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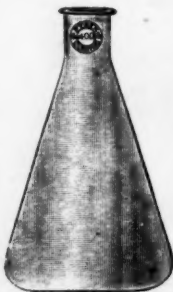
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